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TENSILE PROPERTIES OF PYROLYTIC TUNGSTEN FROM 1370 TO 2980°C IN VACUA

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Pyrolytic tungsten or tungsten deposited from a vapor phase on a heated substrate is a newer form of tungsten, the highest melting metal. Pyrolytic tungsten presents manifold possibilities for shaping because it may be deposited on complex mandrels. It is of interest to know how the pyrolytic material compares with tungsten in other forms. This technical note presents some tensile properties of pyrolytic tungsten from 1370 to 2980°C and makes a comparison with powder metallurgy (PM) tungsten and plasma flame single crystal (PF) tungsten.

Quarter inch plates of pyrolytic tungsten* which became available in the past year were used to make the R-4 type tensile specimens[1] used in this laboratory. Deposition rates from tungsten hexafluoride carried in hydrogen gas were reported by the producer to be from 0.005 to 0.010 in./hr on a graphite substrate held at 650°C. Fig. 1 shows how a specimen was cut from a plate and the orientation of the grains with respect to the tensile axis. Sufficient material was removed to eliminate any possibly contamination from the substrate. This specimen with modified head has a 0.160 in. diameter and 0.640 in. gauge length. Photomicrographs (Fig. 2) of pyrolytic tungsten show a microstructure characteristic of vapor deposited material.

Comparison of the composition of pyrolytic tungsten with PM and PF tungsten is made in Table I. Pyrolytic tungsten

*Material furnished by High Temperature Materials, Inc., Boston, Mass., U. S. A.

has an oxygen and nitrogen content approximately equivalent and a carbon content intermediate to the PM and PF material, all in the "as received" condition. With respect to substitutional impurity elements pyrolytic tungsten exhibits lower values, presumably due to the high purity of the tungsten hexafluoride.

Testing apparatus and procedure have been described.[2] Principal features include an induction heated tungsten susceptor with a hot-grip assembly for holding the specimen on shoulder fillets, and load-elongation recording instrumentation. Vacua employed in this study were of the order of 10^{-4} torr. All testing was carried out at a strain rate of 0.02/min.

Prior to testing, all specimens were heated in vacua to 2845°C for 15 minutes to stabilize the structure for testing up to this temperature. The PM and PF tungsten materials used for comparison were similarly heat treated.[3] In the pyrolytic material the 15 minute treatment resulted in grain growth and the formation of voids (Fig. 3). The anisotropy of the deposit was maintained during grain growth. The volume of voids in the "as heat treated" structure (Fig. 3) is between 5 and 10 per cent as estimated by the line intercept method. The voids may result from fluorine entrapped during deposition.

Typical engineering stress-strain curves are shown in Fig. 4 for the pyrolytic and PM material. Pyrolytic tungsten has lower strength and lower elongation at all test temperatures although the curves show similar shapes at higher temperatures.

Above 1970°C the relatively long region of decreasing stress prior to fracture in both materials is of interest. Although this decrease could possibly be explained by void growth during testing, similar decreasing stress regions were found in the curves for PF tungsten and also for PM tungsten tested at higher strain rates.[3] Neither exhibited void formation or growth.

A summary of the tensile data is given in Figs. 5, 6 and 7. Fig. 5 shows the ultimate tensile strength of pyrolytic tungsten as a function of temperature. The pyrolytic tungsten has lower strength than the PM material at all temperatures and higher strength than the PF material up to 2750°C. The initial void volume in the "as heated treated" pyrolytic tungsten could account for the strength difference relative to PM tungsten. The pyrolytic tungsten even with voids is stronger than the PF tungsten. The lack of grain boundaries in the PF material probably accounts for its lower strength. The relative effect of compositional differences is unknown.

The lower reduction-in-area values for the pyrolytic and PM tungsten as compared to the PF tungsten (Fig. 6) are attributed to the effect of internal voids associated with intercrystalline fracture. In the pyrolytic material this results from the voids present after annealing in combination with void growth during testing. Metallographic studies revealed an increase in void volume with testing. Fracture occurred in the pyrolytic tungsten in an intercrystalline manner at all test temperatures with little or no necking. In Fig. 8 the

microstructure contains a void volume of approximately 18% in a section away from the fracture of a pyrolytic specimen tested at 2200°C. Void growth and intercrystalline fracture were found in the PM material at 1650°C and above.[3] No voids are found in PF material. The predominance of grain boundaries perpendicular to the tensile axis in the pyrolytic material would be expected to increase the tendency for void formation during testing when compared with equiaxed grains of the same grain size in PM material.

Although the volume of voids formed during testing increases with increasing test temperatures the reduction-in-area values remain relatively constant. It is thought that the increase in ductility with increasing temperature in some way offsets the effect of increased void volume.

Elongation data are presented in Fig. 7. Although pyrolytic tungsten has lower values than those for the PM material the shapes of the two curves are similar. PF material shows an increase in elongation throughout the temperature range studied. The slight increase in elongation in the PM material above approximately 2200°C was attributed to stress-induced grain growth.[3] Although little or no grain size change is observed in the pyrolytic material during testing, it exhibits a slight increase in elongation above 2200°C followed by a decrease above 2480°C. Reasons for lower elongation values above approximately 2500°C for pyrolytic tungsten and above approximately 2750°C for PM tungsten are unknown.

Although insufficient pyrolytic material was available for extensive testing, a limited study of the formation of voids

resulting from high temperature heating was conducted. Void formation has been reported in PM tungsten wire rapidly heated above 2850°C in vacuum.[4] A vacuum pretreatment at lower temperatures was found to eliminate the voids on subsequent heating to higher temperature. Similarly, heating a pyrolytic tungsten specimen to 2200°C produced a microstructure containing no voids and with grains of approximately the same diameter (0.14 mm) as resulted from the 2845°C heat treatment.

One specimen, with a slightly modified gauge diameter, was given a 2200°C anneal and tested at 1930°C. Data are plotted with a filled triangle symbol (▲) in Figs. 5, 6 and 7 to compare with pyrolytic tungsten annealed at 2845°C. Tensile strength (Fig. 5) is seen to be slightly higher presumably due to the absence of voids in the specimen prior to testing. Fracture occurred in an intercrystalline mode and voids were observed in the microstructure after testing. Reduction-in-area and elongation values (Figs. 6 and 7) are found to be unchanged or even slightly lower. This suggests, on the basis of one test, that voids present after the high temperature anneal have a relatively minor effect on ductility compared with voids formed during testing. As previously noted, the presence of grain boundaries perpendicular to the tensile axis appears to enhance the sensitivity to void formation and intercrystalline fracture.

In PM materials detrimental substitutional impurities such as iron have been singled out as being a possible cause of void nucleation and growth.[5] This pyrolytic material even with low substitutional impurity levels still shows void formation

and growth. However grain boundary orientation, and possibly entrapped fluorine may have some overriding effect.

It should be emphasized that these data are for one limited lot of pyrolytic tungsten. Tungsten deposited at other rates and temperatures may have different properties as varying conditions may be expected to affect grain size, and to a lesser extent, impurity level.

FIGURE CAPTIONS

Fig. 1. Specimen orientation in pyrolytic tungsten.

Fig. 2. Pyrolytic tungsten "as deposited". Etched 1 minute in 30 cc lactic acid, 10 cc HNO_3 and 10 cc HF. (X100)

(a) Section parallel to deposition plane.

(b) Section normal to deposition plane showing graphite substrate.

Fig. 3. Pyrolytic tungsten annealed 10 minutes at 2845°C. Etched 1 minute in 30 cc lactic acid, 10 cc HNO_3 and 10 cc HF. (X100)

(a) Section parallel to deposition plane.

(b) Section normal to deposition plane.

Fig. 4. Typical engineering stress-strain curves at indicated temperatures (°C) for pyrolytic and powder metallurgy tungsten.

Fig. 5. Ultimate tensile strength of pyrolytic tungsten.

Fig. 6. Reduction in area of pyrolytic tungsten.

Fig. 7. Elongation of pyrolytic tungsten.

Fig. 8. Pyrolytic tungsten tested at 2200°C. Etched 1 minute in 30 cc lactic acid, 10 cc HNO_3 and 10 cc HF. (X100)

(a) Section parallel to deposition plane.

(b) Section normal to deposition plane.

TABLE I

CHEMICAL COMPOSITION OF TUNGSTEN MATERIALS

	Concentration (p.p.m.)					
	Pyrolytic				Powder Metallurgy (PM)	Plasma Flame (PF)
Element	As Received*	2000°C Anneal	As Received	2845°C Anneal	As Received	As Received
Oxygen	12	12	4.9	3.2	1.3-5	5-9
Carbon	17	17	31.0	15.4	2-4	66-98
Nitrogen	10	10	4.0	4.0	5-16	3-7
Hydrogen	2	2	--	--	--	--
Fluorine	10	10	--	--	--	--
Silicon	10	1	10	--	10-20	20-40
Iron	10	10	10	--	10-40	--
Molybdenum	ND	ND	10	--	30-50	--
Aluminum	10	1	10	--	20-40	10-30

* Representative analyses supplied by High Temperature Materials, Inc. Other analyses from a commercial laboratory.

ND = Not Detected

-- = Not Determined

REFERENCES

1. Federal Test Method Standard No. 151, "Metals: Test Methods" General Services Administration, Business Service Center, Washington, D. C., July 1956.

2. J. L. Taylor, submitted to Rev. Scien. Inst. October 1962.

3. J. L. Taylor and D. H. Boone, submitted to ASM Trans., April 1963.

4. H. Cline and D. P. Ferris, Trans. AIME, 224 (1962) 632.

5. H. G. Sell, G. H. Keith, R. C. Koo, and R. Corth, Aeronautical Systems Division, Wright Patterson AFB, Ohio, Technical Report No. WADD-TR-60-37 (1962) 9.

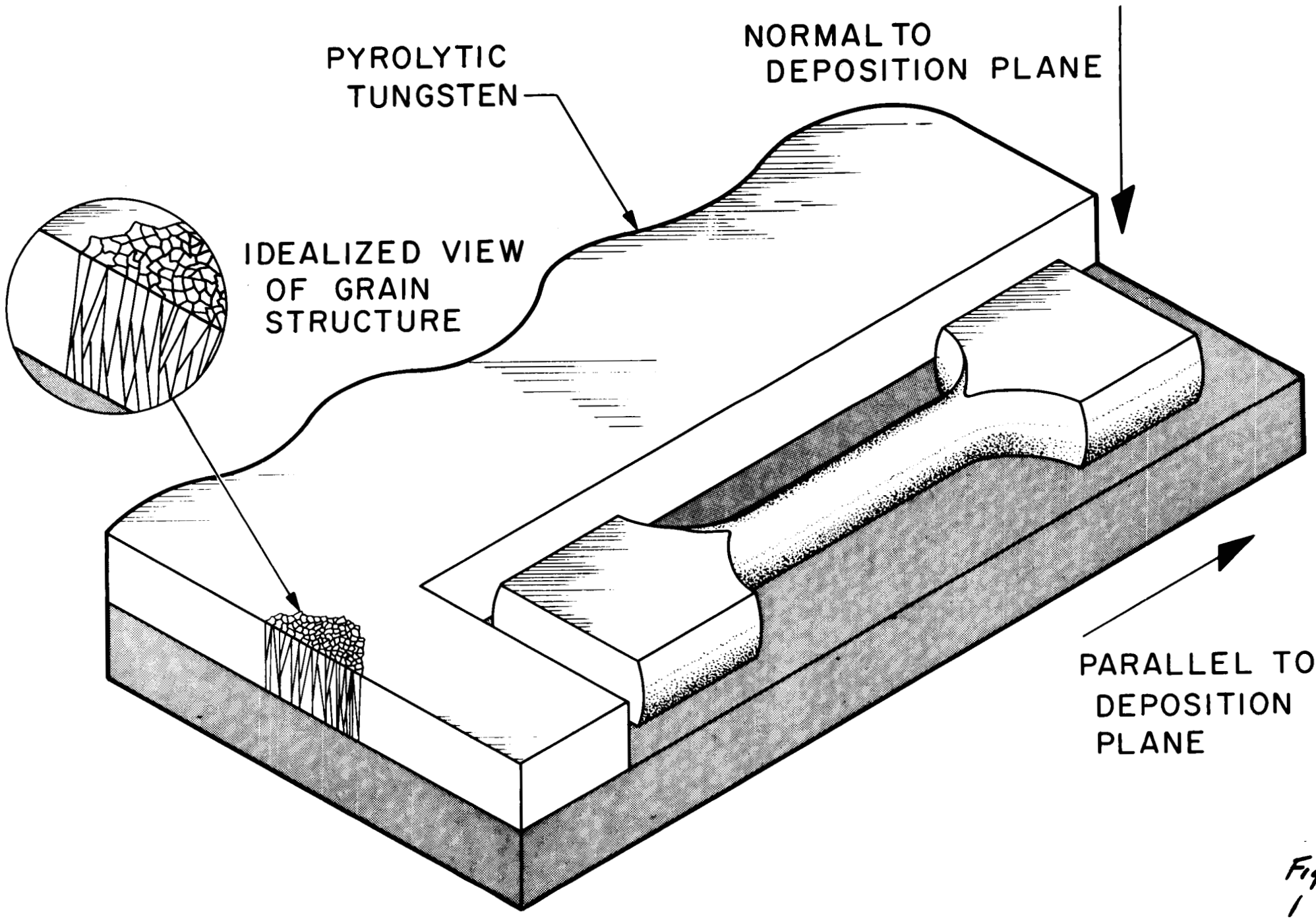
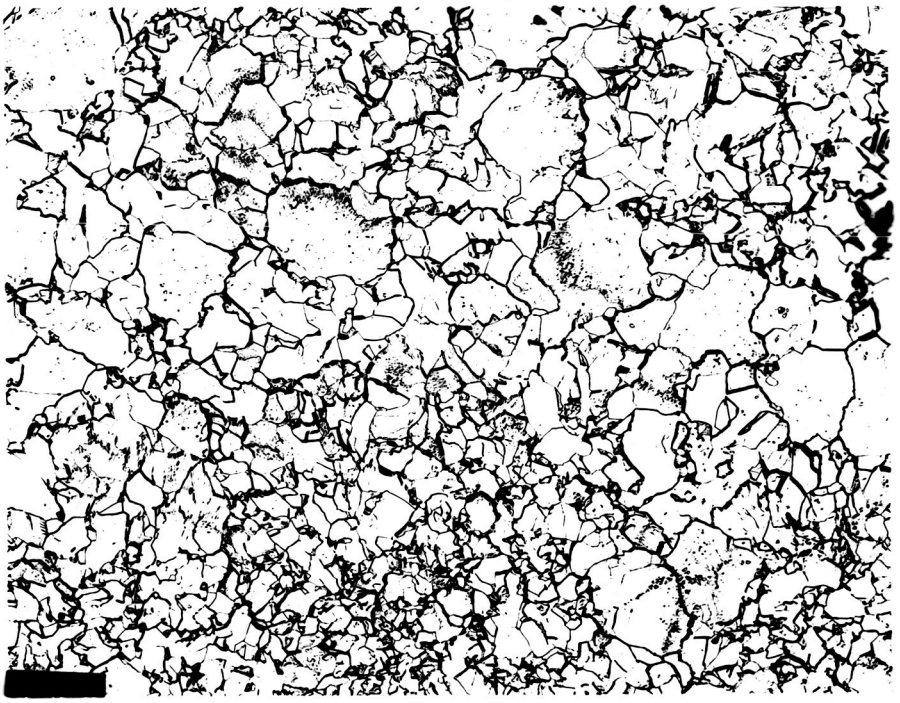

PYROLYTIC
TUNGSTEN
NORMAL TO
DEPOSITION PLANE
IDEALIZED VIEW
OF GRAIN
STRUCTURE
PARALLEL TO
DEPOSITION
PLANE

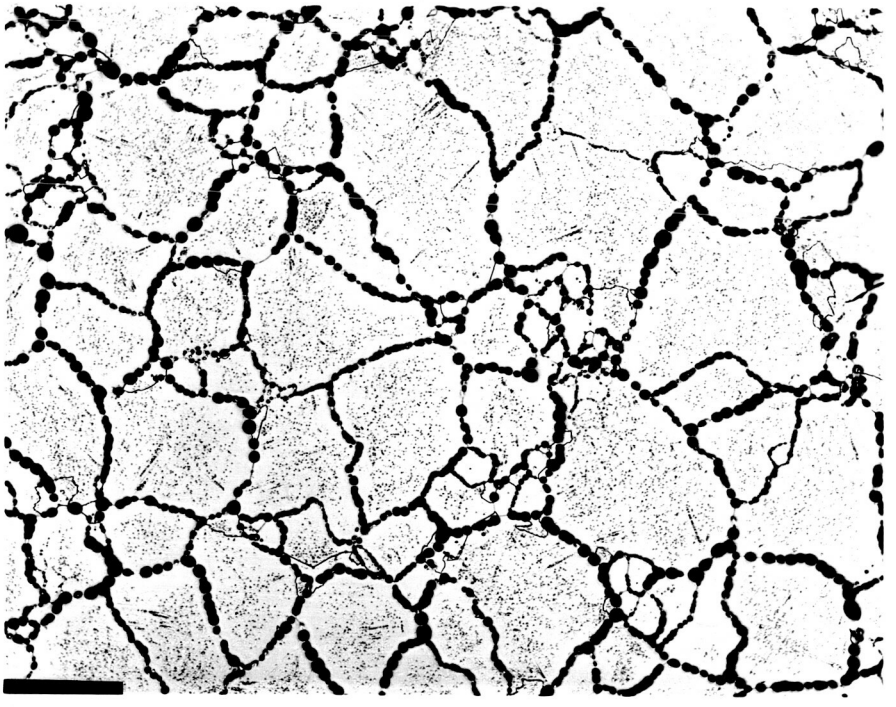
Fig. 1



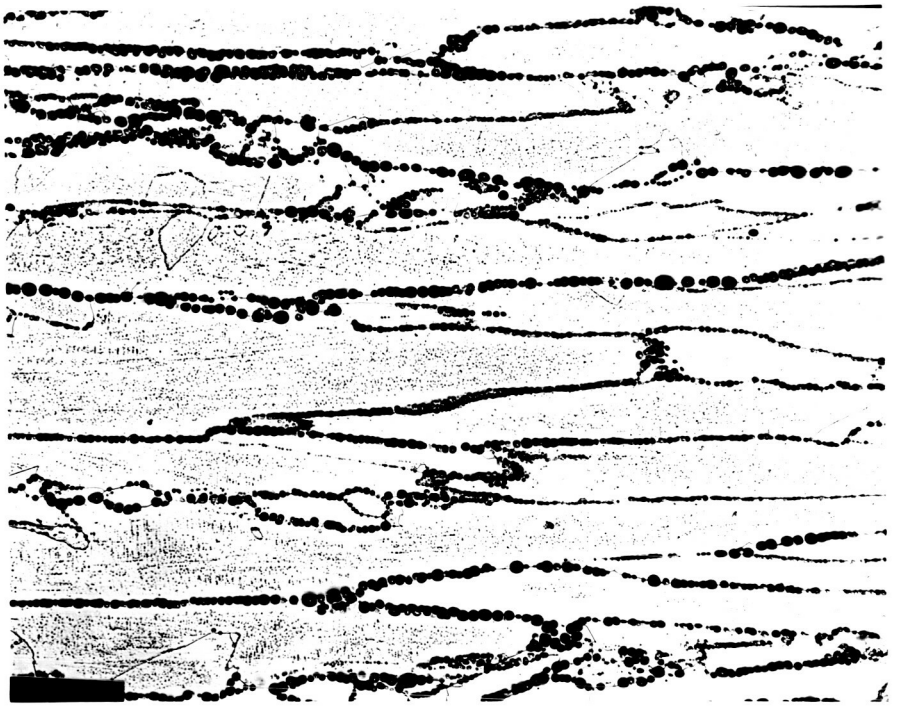
a.

b.

Fig. 2



a.



b.

Fig. 3

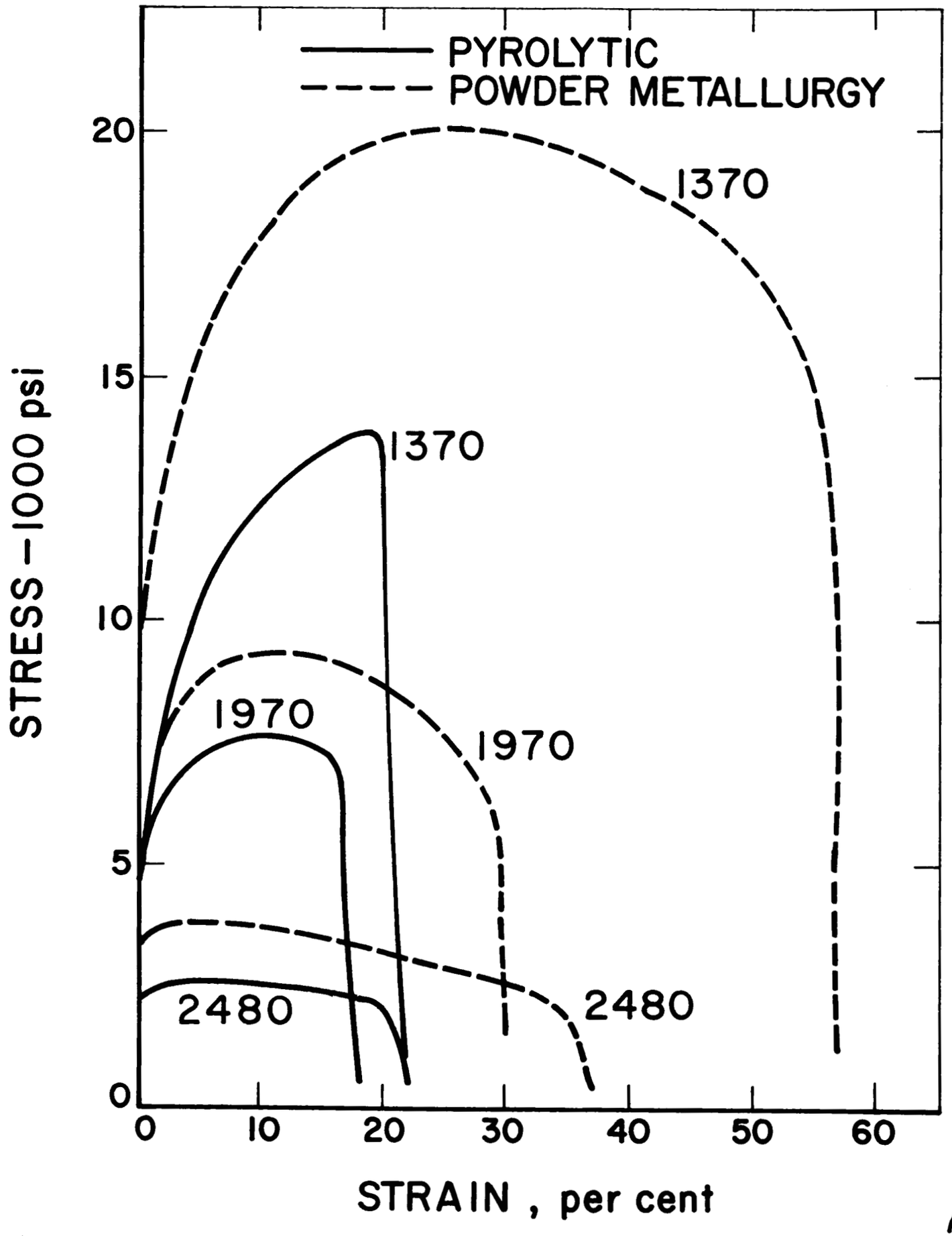

PYROLYTIC
POWDER METALLURGY
20
15
10
5
0
1370
1370
1970
1970
2480
2480
0
10
20
30
40
50
60
STRESS – 1000 psi
STRAIN , per cent

Fig 4

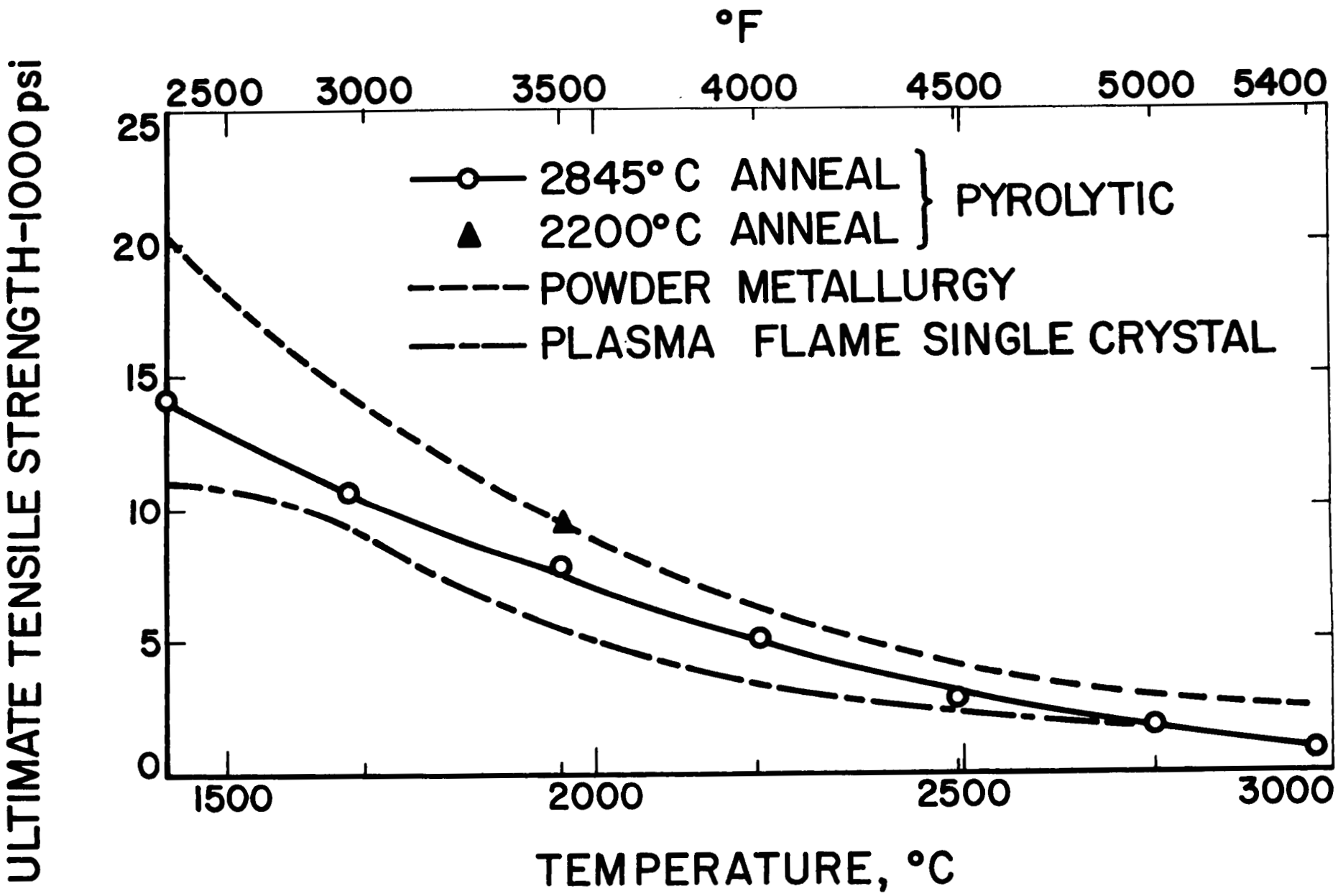
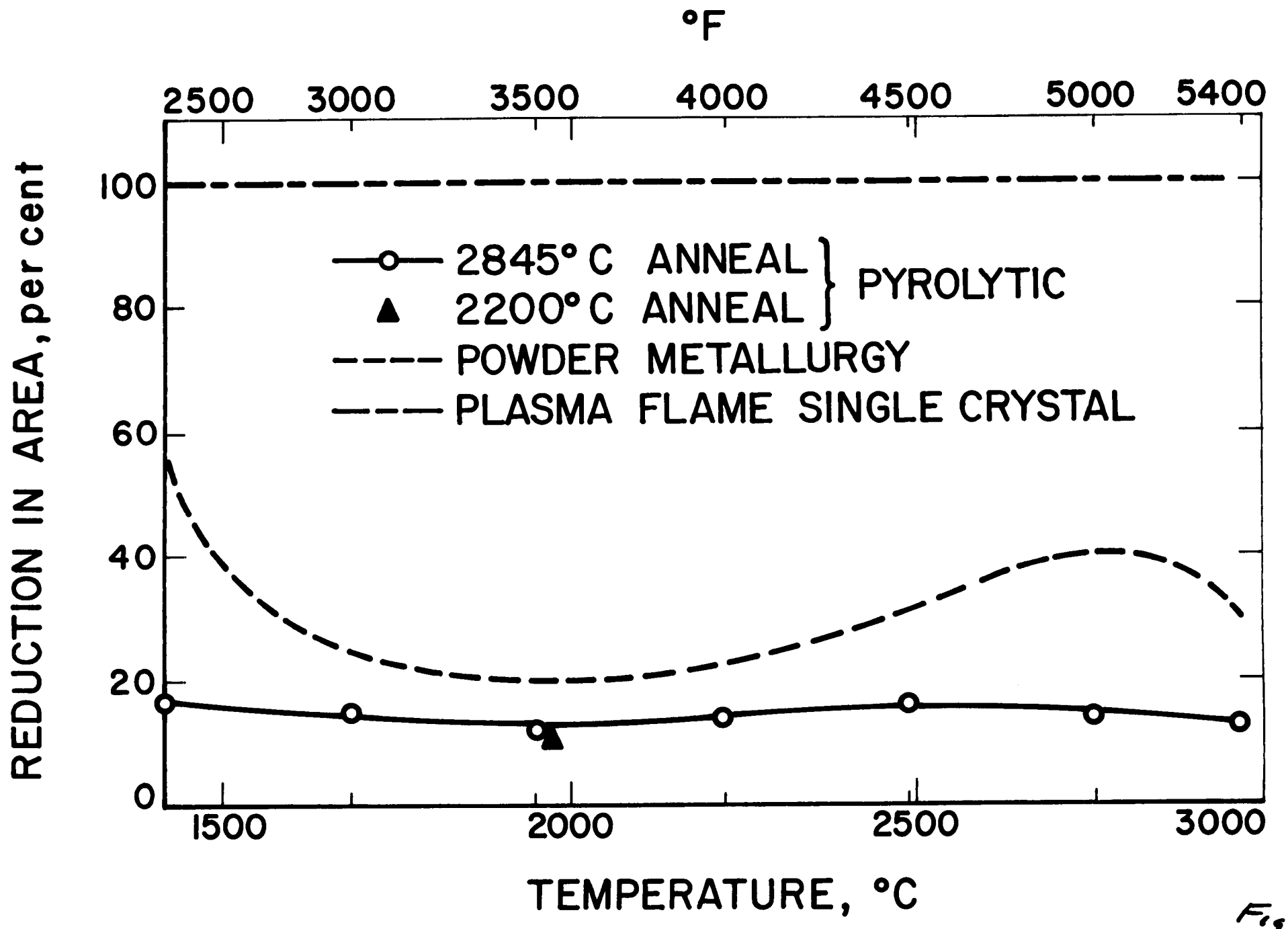

°F
2500
3000
3500
4000
4500
5000
5400
25
20
15
10
5
0
2845° C ANNEAL
2200°C ANNEAL
PYROLYTIC
POWDER METALLURGY
PLASMA FLAME SINGLE CRYSTAL
ULTIMATE TENSILE STRENGTH-1000 psi
1500
2000
2500
3000
TEMPERATURE, °C

Fig 5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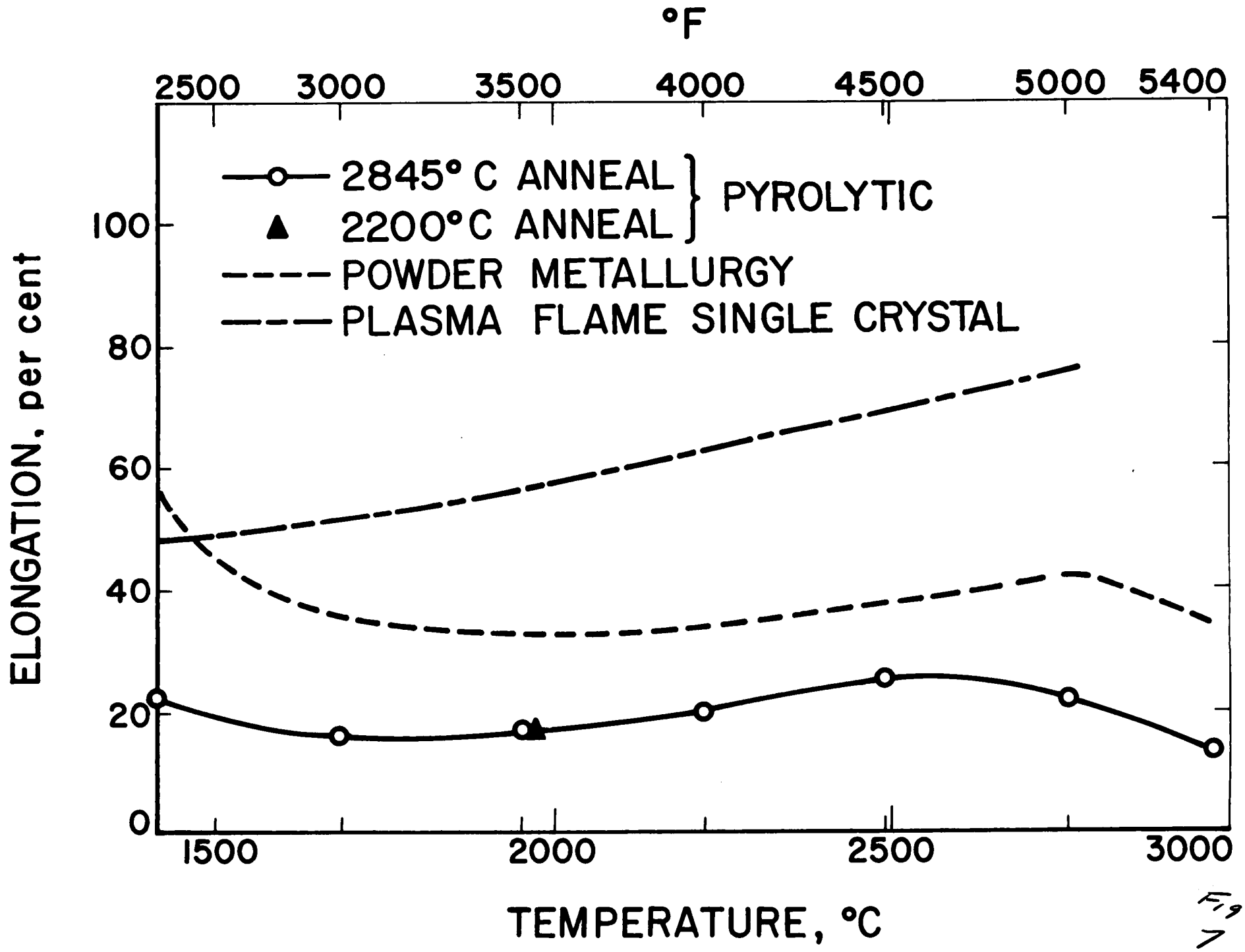
0
2845° C ANNEAL
2200° C ANNEAL
PYROLYTIC
POWDER METALLURGY
PLASMA FLAME SINGLE CRYSTAL
REDUCTION IN AREA, per cent
1500
2000
2500
3000
TEMPERATURE, °C

Fig 6


°F
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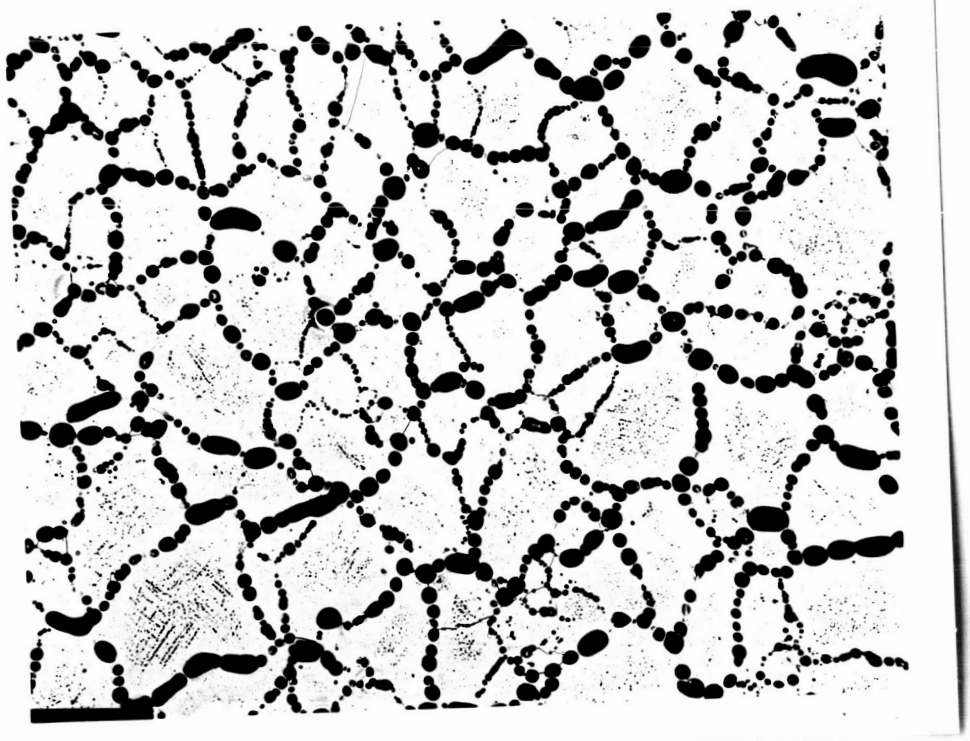
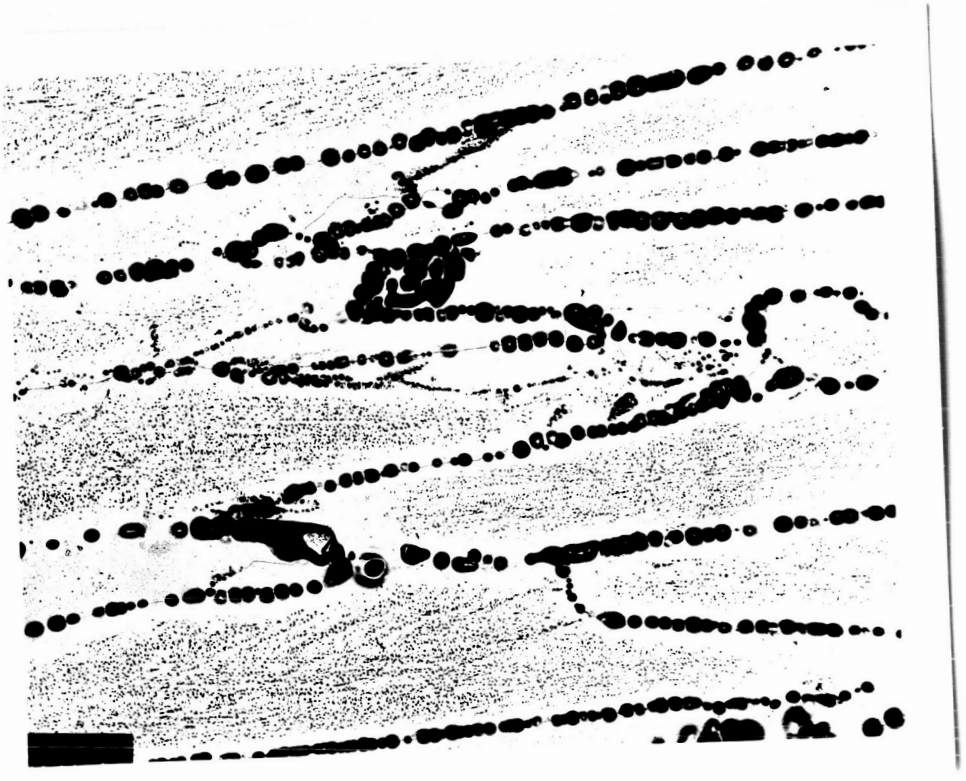
2845° C ANNEAL
2200° C ANNEAL
PYROLYTIC
POWDER METALLURGY
PLASMA FLAME SINGLE CRYSTAL
ELONGATION, per cent
100
80
60
40
20
0
1500
2000
2500
3000
TEMPERATURE, °C

Fig 7



a.



b.

Fig. 8